

Development of bakelite based Resistive Plate Chambers

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Abstract

A Comparative study has been performed on Resistive Plate Chambers made of different grades of bakelite paper laminates, produced and commercially available in India. The chambers, operated in the streamer mode using argon : tetrafluoroethane : isobutane in 34:59:7 mixing ratio, are tested with cosmic rays for the efficiency and the stability with cosmic rays. A particular grade of bakelite (P-120, NEMA LI-1989 Grade XXX), used for high voltage insulation in humid conditions, was found to give satisfactory performance with stable efficiency of $> 96\%$ continuously for more than 110 days. A silicone treatment of the inner surfaces of the bakelite RPC is found to be necessary for operation of the detector.

Key words: RPC; Streamer mode; Bakelite; Cosmic rays

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1. Introduction

The Resistive Plate Chambers (RPCs), first developed by Santonico et al. [1] using bakelite and by Yu. N. Pestov et al. [2, 3] and subsequently by others [4], using silicate glass are used extensively in high energy physics experiments. The RPCs are being considered for the following reasons a) relatively low cost of materials used in making RPCs, b) robust fabrication procedure and handling and c) excellent time and position resolution. Primarily used for generating faster trigger for muon detection [5], time of flight (TOF) [6, 7] measurement, and tracking capabilities in multi layer configurations, they are successfully used in BELLE [8], BaBar [9], BESIII [10], and several upcoming LHC experiments (ATLAS, CMS etc.) [11, 12]. RPCs are used in neutrino experiments like OPERA where its excellent time resolution and tracking capabilities are exploited [13]. The RPCs are also being explored for use in PET imaging with TOF-PET [14], detection of γ -rays [15] and neutrons [16, 17] over a large area.

The RPCs are made up of high resistive plates (e.g. glass, bakelite, ceramics etc.) as electrodes, which help to contain the discharge created by the passage of a charged particle or an ionizing radiation in a gas volume, and pick-up strips are used to collect the resulting signals. Typical time resolution for a single gap RPC is $\sim 1\text{-}2$ ns. By reducing the gaps between the electrodes or by using multi-gap configuration, time resolution in such a detector can be reduced to < 100 ps [18, 19].

The RPCs are operated in two modes, viz., the proportional mode and the streamer mode [20]. Over the years, one of the main concerns with the use of RPCs is their long term stability. In the proportional mode, a small amount of charge is produced in the gas, which allows the RPC to recover in a relatively shorter time to handle high counting rates (~ 1 KHz/cm²). Ageing effects caused by the accumulated charge is also relatively less in this mode. In the streamer mode, the amount of charge produced is considerably larger creating induced signals of larger magnitude. But, the recovery time is larger and the irreversible damage caused by the accumulated charge reduces the life of the RPC. However, several remedial measures can be taken to prolong its life under streamer mode of operation. Careful choice of materials, smoothness of the surfaces to avoid localization of excess charges, surface treatment to reduce the surface resistivity or providing alternate leakage path for post-streamer recovery are adopted in the major high energy physics experiments. Prolonged stable operation in streamer mode of the BELLE RPCs, though made of glass, is a testimony to many serious efforts taken for the above cause [8].

The glass-based RPCs are found to be more stable mainly for low rate applications, even though some erosion effects are found for such cases, particularly when these are operated in the streamer mode [20]. This has been attributed to the corroding of the glass surface due to the large charge build up in the streamer mode of operation. However, in the proportional mode of operation, the detectors can be operated for longer period. At the end of nineties, it was found that the RPCs based on bakelites show serious ageing effects reducing the efficiency drastically [21]. Detailed investigations revealed that the use of linseed oil for the surface treatment in such cases was the main reason for this ageing effect [22, 23]. Efforts were subsequently made to look for alternatives to linseed oil treatment, or to develop bakelite sheets which can be used without the application of linseed oil [24]. It has, however, been found that for several ongoing and future applications (e.g. CMS detectors), bakelite based RPCs are chosen as preferred options mainly due to cheaper cost of fabrication.

In the proposed India-based Neutrino Observatory (INO), the RPCs have been chosen as the prime active detector for muon detection in an Iron Calorimeter (ICAL) [25]. As proposed presently, ICAL is a sampling calorimeter consisting of 140 layers of magnetized iron, each of 60 mm thickness, using RPCs of $2\text{m} \times 2\text{m}$ area as active media sandwiched between them. A 50 Kton ICAL is expected to consist of about 27000 RPC modules. For ICAL RPCs, main design criteria are (a) good position resolution, (b) good timing resolution (c) ease of fabrication in large scale with modular structure and most importantly (d) low cost. Detailed R & D are being performed on glass RPCs for this application.

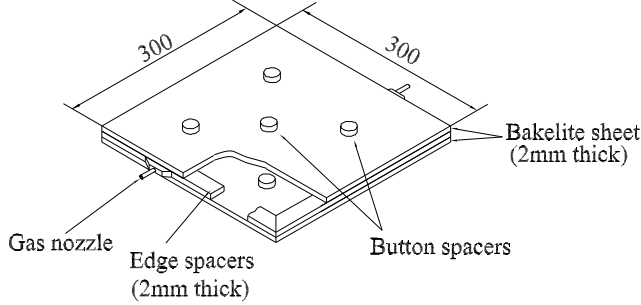


Figure 1: Schematic diagram of a resistive plate chamber.

In this article, we report a parallel effort on building and testing of the RPC modules using the bakelite obtained from the local industries in India. The aim of the study is to achieve stable performance of the RPC detector for prolonged operation.

The paper is organized as follows. In the next section, we describe the method of assembly of the RPC modules. The section 3 contains measurements of the bulk resistivity of the bakelite sheets used in this application while the cosmic ray set-up used in our experiment are discussed in section 4. The results of the study are reported in section 5.

2. Construction of the RPC modules

A schematic view of the assembled RPC modules is shown in the Fig. 1. Two $300 \text{ mm} \times 300 \text{ mm} \times 2 \text{ mm}$ bakelite sheets are used as electrodes. The inner surfaces of the two sheets are separated by a 2 mm gap. Uniform separation of the electrodes are ensured by using five button spacers of 10 mm diameter and 2 mm thickness, and edge spacers of $300 \text{ mm} \times 8 \text{ mm} \times 2 \text{ mm}$ dimension, both being made of polycarbonate. Two nozzles for gas inlet and outlet, also made of polycarbonate, are placed as part of the edge spacers. All the spacers and nozzles are glued to the bakelite sheets using Araldite[®] epoxy adhesive (grade: AY 103**MP, made by Huntsman A.M., (Europe)). The 2 mm thick active gas gap of the RPC modules are leak-checked using argon and helium sniffer probes. The edges of the bakelite sheets are sealed by applying a layer of the epoxy adhesive to prevent permeation of moisture.

After proper cleaning, a graphite coating is made on the outer surfaces of bakelite sheets to distribute the applied voltage uniformly over the entire RPC. A gap of 10 mm from the edges to the graphite layer is maintained to avoid external sparking. The surface resistivity varies from $500 \text{ K}\Omega/\square$ to $1 \text{ M}\Omega/\square$ for different samples. The graphite coating, applied by using a spray gun, however, results in a non-uniformity (less than 20%) for a particular coated surface. Two

small copper foils $\sim 20 \mu\text{m}$ thick are pasted by kapton tape on both the outer surfaces for the application of high voltage. The HV connectors are soldered on these copper strips. Equal HVs are applied on both the surfaces.

In order to collect the accumulated induced charges, pick-up strips are placed above the graphite coated surfaces with minimum air-gap. The pick-up strips are made of copper ($20 \mu\text{m}$ thick), pasted on one side of 10 mm thick foam. The area of each strip is $300 \text{ mm} \times 30 \text{ mm}$ with a separation of 2 mm between two adjacent strips. The pick-up strips are covered with $100 \mu\text{m}$ thick kapton foils to insulate them from the graphite layers. The ground plane, made of aluminium, is pasted on the other side of the foam. The signals from different strips are sent through a ribbon cable, followed by RG-174/U coaxial cables using proper impedance matching.

The gases used in the RPC are mixtures of Argon, Isobutane and Tetrafluoroethane (R-134a) in varying proportion. The gases are pre-mixed, stored in a stainless steel container and sent to the detector using stainless steel tubes. A typical flow rate of 0.4 ml per minute resulting in ~ 3 changes of gap volume per day is maintained by the gas delivery system. A systematic analysis was made for R-134a and Isobutane before use in the system by a Prisma Quadstar 422 Residual Gas Analyzer. The composition was found to be 98.83% for R-134a with 0.75% O_2 and 0.41% N_2 and 98.93% for Isobutane with 1.07% H_2 .

3. Measurement of bulk resistivity of bakelite

The bakelite sheets are phenolic resin bonded paper laminates. In the present work, three types of bakelite sheets have been used to build as many modules. They are (a) mechanical grade bakelite (P-1001), (b) Superhylam grade and (c) electrical grade (P-120).

The P-1001 and P-120 grade bakelites are manufactured by Bakelite Hylam, India and the Superhylam grade is obtained from the other manufacturer Super Hylam, India. The sheets of P-1001 and P-120 are matt finished whereas superhylam is glossy finished. The P-1001 has good mechanical properties whereas the P-120 has good mechanical and electrical properties under humid conditions prevalent in India.

The bulk resistivity of the electrode plates of the RPC is an important parameter [26]. The high resistivity helps in controlling the time resolution, singles counting rate and also prevents the discharge from spreading through the whole gas. We have measured the bulk resistivities of the bakelite sheets via the measurement of the leakage current.

This measurement is performed at the same place and the same environment where the RPCs have been tested. The test set up is kept in a temperature and humidity controlled room. These two parameters have been monitored during the experiment and are nearly the same around that time. The bulk resistivities of different grade materials at 4 kV are tabulated in Table 1. The volume resistivity(ρ) vs. voltage(V) characteristics of different grade materials are shown in Fig. 2. It is clear from the figure that the bulk resistivity is considerably higher for the P-120 grade bakelite. For the P-1001 grade, resistivity

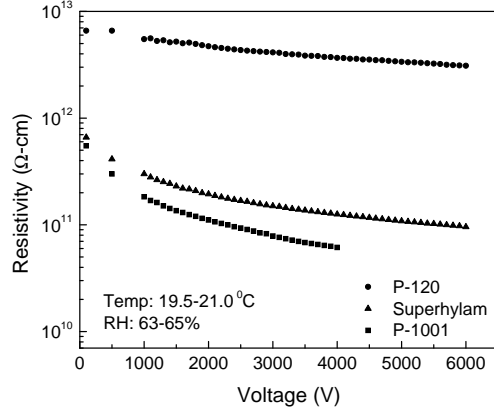


Figure 2: The volume resistivity (ρ) as a function of the applied voltage for three grades of bakelites.

Table 1: Mechanical and electrical properties of different grades of bakelite.

Trade Name	NEMA LI-1989 Grade	BS-2572 Grade	Density (g/cc)	Electrical strength (kV/mm)	Surface finish	Bulk resistivity ($\Omega\text{-cm}$)
P-1001	X	P1	1.38	3.5	Matt	6.13×10^{10}
Superhylam	-	P2	1.72	9.5	Glossy	1.25×10^{11}
P-120	XXX	P3	1.22	9.5	Matt	3.67×10^{12}

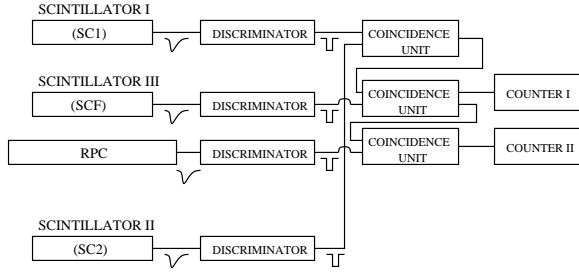


Figure 3: Schematic representation of the cosmic ray test setup.

is much lower and it cannot sustain high voltage above 4 kV. Therefore, the P-1001 grade is not considered further for building up the RPC. The superhy-lam grade, though having lower bulk resistivity than P-120, would stand high voltages up to 6 kV. This is also considered for the fabrication of RPC detector.

4. Cosmic ray test setup

Fig. 3 shows the schematic of the setup for testing the RPC modules using cosmic rays. Three scintillators, two placed above the RPC plane and one placed below are used for obtaining the trigger from the incidence of the cosmic rays. The coincidence between scintillator I (350 mm \times 250 mm size), scintillator II (350 mm \times 250 mm size) and the finger scintillator(III) (200 mm \times 40 mm size) is taken as the Master trigger. Finally, the signal obtained from the pick-up strip of the chamber is put in coincidence with the master trigger obtained above. This is referred to as the coincidence trigger of the RPC. The width of the finger scintillator is made smaller than the total width of the two adjacent readout strips, thereby needing a correction for dead zones in between two readout strips.

The high voltage to the RPC, are applied at the ramping rate of 5 V/s on both the electrodes. The streamer pulses are obtained starting from the high voltage of 5 kV across the RPC. The high voltage is applied to some of the RPCs by using the CAEN Mod.N470 unit and to the others using the CAEN Mod.N471A unit. The leakage current as recorded by the high voltage system is studied. The signals from two consecutive strips covered by the scintillator III are ORed to form the final signal, for the next part of the pulse processing.

The Philips Scientific leading edge discriminators (Model 708) are used for the scintillators and the RPC pulses. Various thresholds are used on the discriminators to reduce the noise. For our final results, a threshold of 40 mV is used on the RPC signal. We have used a CAMAC-based data acquisition system LAMPS, developed by Electronics Division, Bhabha Atomic Research Centre, Trombay, India. Counts accumulated in a CAEN (Model 257) scalar over a fixed time period are recorded at regular intervals, and saved in a periodic

log database. The temperature and the humidity are monitored at the time of measurement.

5. Results

An important and obvious goal of any RPC detector development is to study the long term stability with high efficiency. In that spirit, the following studies are performed in the cosmic ray test bench of the RPC detectors.

The efficiency of the RPC detector, taken as the ratio between the coincidence trigger rates of the RPC and the master trigger rates of the 3-element plastic scintillator telescope as mentioned in sec.4, is first studied by varying the applied HV for each detector. The rates are calculated from data taken over 30 minutes duration for each HV setting. The temperature and humidity during these measurement are recorded to be about 22-25°C and 63-65% respectively. The average master trigger rate is $\simeq 0.005 \text{ Hz/cm}^2$. The variation of efficiency with applied HV is shown in the Fig. 4 and that of the singles counting rates with the HV is shown in the Fig. 5. It is seen that for both the bakelite grades, the efficiency has increased from 20% to 75% as HV is ramped up from 6.5 kV to 6.8 kV. The efficiency for the superhylam grade gradually increases and reaches the plateau at $\sim 96\%$ from 7.5 kV, while that of the P-120 grade reaches a maximum of $\sim 79\%$ at 7.2 kV and then decreases steadily up to $\sim 35\%$ as the HV is increased to 9 kV. The singles counting rates in both the cases, however, have increased more or less exponentially with sudden jumps around 6.5-7.0 kV (see Fig. 5), i.e. near the points where the efficiency becomes uniform (in case of superhylam) or starts to decrease (in case of P-120). This possibly indicates the onset of a breakdown regime that recovers in a reasonable time for the superhylam grade RPC but works the other way for the P-120 grade bakelite RPC. It should, however, be noted that the singles counting rate and the leakage current of the superhylam RPC are both larger than those of the P-120 RPC, which are expected on the basis of smaller bulk resistivity of the superhylam grade bakelite.

In order to investigate the reason for the above phenomena, and taking cue from the fact that superhylam surfaces are glossy finished while the P-120 surfaces are matt finished, we have dismantled the detectors and made surface profile scans over a 5 mm span of the surfaces using DekTak 117 Profilometer. These scans, done also for the P-1001 grade, are shown in Fig. 6. It is clearly seen that the three surfaces have a short range variation (typically $\sim 0.1 \mu\text{m}$ length scale) and a long range variation (typically $\sim 1 \mu\text{m}$ length scale). The long range surface fluctuation, which is a measure of non - uniformity, averaged over several scans are: $0.84 \pm 0.12 \mu\text{m}$ (P-120), $0.49 \pm 0.17 \mu\text{m}$ (superhylam) and $0.88 \pm 0.09 \mu\text{m}$ (P-1001). Thus the long range fluctuations, within the limits of experimental uncertainties, are nearly the same. On the other hand the short range fluctuations, a measure of surface roughness, are: $0.64 \pm 0.06 \mu\text{m}$ (P-120), $0.17 \pm 0.02 \mu\text{m}$ (superhylam), and $0.63 \pm 0.13 \mu\text{m}$ (P-1001), and thus indicate a superior surface quality of the superhylam grade.

To explore a remedial measure, we have applied a thin layer of viscous silicone

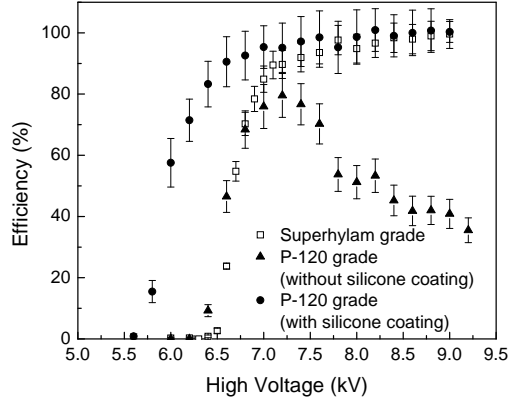


Figure 4: The efficiency as a function of high voltage for two RPCs (silicone coated & uncoated P-120 and Superhylum grade bakelite) obtained with a gas mixture of Argon (34%) + Isobutane (7%) + R-134a (59%). The threshold for the RPCs are set at 40 mV for P-120 and 50mV for superhylum.

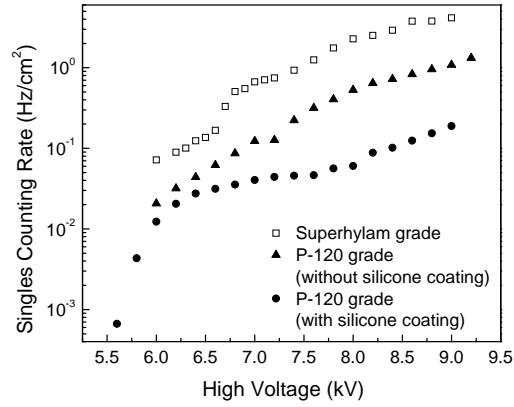


Figure 5: Singles counting rate as a function of high voltage.

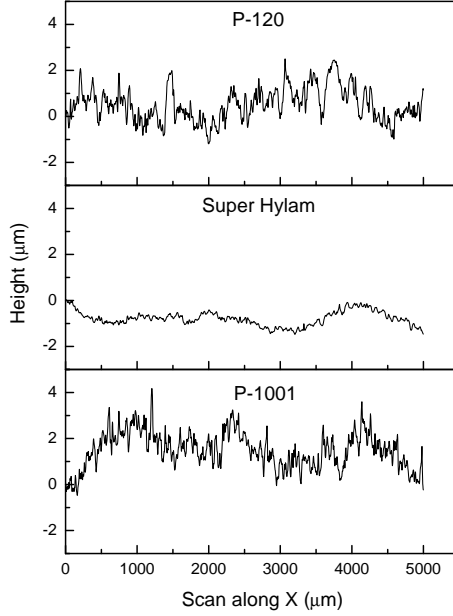


Figure 6: Linear surface profile scans of the three grades of bakelite sheets.

fluid (chemical formula : $[R_2SiO]_n$, where R = organic groups such as methyl, ethyl, or phenyl) [coefficient of viscosity = 5500 cP, manufactured by Metroark Limited, Kolkata, India] on the inner surfaces of the P-120 bakelite sheets. About 1 gm of the fluid is applied over 300 mm \times 300 mm area. Based on the specific gravity (1.02 at 23°C) of the fluid, the estimated coating thickness would be $\sim 10 \mu\text{m}$. This material is chosen for the following reasons: a) very low chemical reactivity with the gases used; b) good thermal stability over a wide temperature range (from -100 to 250 °C); c) very good electrical insulator; d) excellent adhesion to most of the solid materials, and e) low vapour pressure, which is essential for stable operation over a reasonable time period. The silicone treated surfaces are kept under infrared lamp for 24 hours to allow the viscous fluid to fill all the micro-crevices on the surface. The reassembled detector is tested at the same set-up. The results of efficiency and singles count rate measurements, shown in the Figs. 4 and 5, indicate a remarkable improvement in the performance of the P-120 detector. The efficiency reaches from 20% to 75% as the HV is increased from 5.7 kV to 6.2 kV, while the singles count rate, as a whole has decreased by a factor of 5. This indicates quenching of micro-discharge after silicone treatment, which is very much desirable for functioning of the detector. The efficiency in this case reaches $> 95\%$ plateau at 7 kV.

It is worth noting that surface treatment with insulating / non-polar liquid as a remedial measure was first demonstrated for the BaBar RPCs. However,

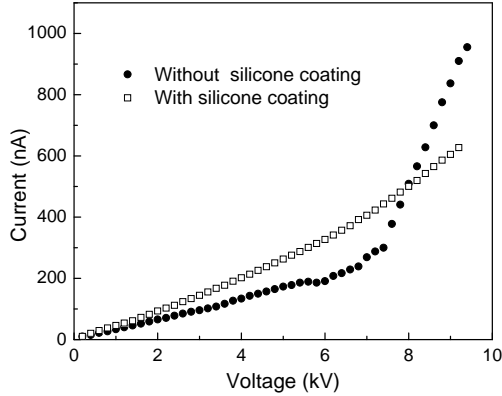


Figure 7: Current as a function of the applied voltage for RPC made by P-120 grade bakelite.

formation of stalagmites by polymerisation of uncured linseed oil droplets had created conducting paths through the gap, thereby causing irreversible damage to the bakelite plates [22]. The process of linseed oil treatment was later changed by increasing the proportion of eptane as a thinner to produce a thinner coating (10-30 μm) on the inner surface [27]. Our observation that silicone coating of the inner surfaces aids the proper functioning of our P-120 bakelite RPC detector once again confirms the importance of smooth surface finish of the inner surfaces.

To judge the improvement in the overall performance of the RPC detector, we have measured the leakage current through the RPC detector with and without silicone coating and the plot of these as a function of the applied HV as shown in the Fig. 7. Both the plots show a common feature that the current-voltage curves have two distinctly different slopes. While the gas gap behaves as an insulator in the lower range of applied voltage and hence the slope over this span scales as the conductance of the polycarbonate spacers, at higher range of voltage, the gas behaves as a conducting medium due to the formation of the streamers. Therefore, the slope over this range scales as the conductance of the gas gap. It is seen that the slope in the higher range of voltage is much steeper for the RPC without silicone coating and hence it points to the fact that some sort of uncontrolled streamers are being formed in the gas gap causing a degradation of the efficiency. This possibly does not happen in the RPC detector with silicone coating.

We have also examined the effect of discriminator threshold setting on the efficiency curves of the RPC with silicone treated surfaces. These are plotted in the Fig. 8. It is clear that the efficiency curves do not depend much on the threshold setting from 20 mV to 80 mV, except that the efficiency plateau is marginally higher at the lowest threshold setting of 20 mV.

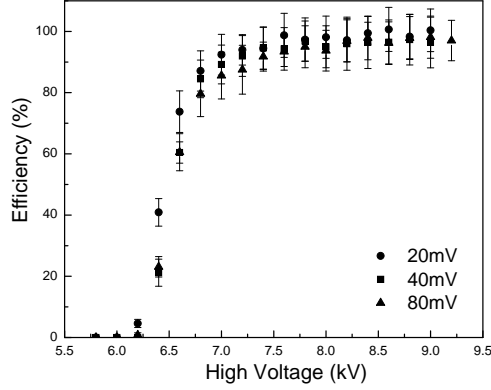


Figure 8: Efficiency versus high voltage for different thresholds for silicone coated P-120 grade bakelite RPC with silicone coating.

The effect of humidity on the efficiency curves has also been studied. This measurement has been done at relative humidities of 58% and 67% of the laboratory environment and at the same room temperature of $\sim 23^\circ\text{C}$. These curves, plotted in the Fig. 9, indicate no effect of humidity on the efficiency. However, the leakage currents, measured simultaneously and plotted in the Fig. 10, are a bit larger at higher humidity. This observation indicates that charge leakage through the exterior surfaces may be contributing more at higher humidity.

The long term stability of the bakelite RPCs has been studied using the same cosmic ray test set-up. The coincidence trigger counts of the RPCs and the master trigger counts, accumulated over every 2 hours, have been recorded continuously for more than 6 months. The room temperature and the relative humidity have been controlled to keep them less than 24°C and 80%, respectively. When the humidity was larger, particularly during the monsoon season, the test set-up was shut down till it came down to 80% and below. The singles count rates of the RPCs have also been recorded simultaneously. The Figs. 11 and 12 depict the variation of efficiency and singles count rates over the above mentioned period for both the grades of RPCs. The superhylum grade RPC has worked with an efficiency of $> 95\%$ which remained steady for 25 days, but beyond that, it deteriorated gradually to $\sim 86\%$ efficiency within next 13 days. The singles count rate, however, has increased from day one from 1 Hz/cm^2 to 10 Hz/cm^2 within 10 days, and then it increased slowly over the next 28 days. After that period, the singles count rate shot up to $> 30 \text{ Hz/cm}^2$. The leakage current gradually increased from $3\text{-}4 \mu\text{A}$ to $> 10 \mu\text{A}$ within that period. This RPC was discontinued after 38 days and the silicone coated P-120 grade RPC was then mounted. The efficiency measured was $\sim 96\%$ and above and has remained steady for more than 110 days. The singles count rate also has remained steady around 0.1 Hz/cm^2 . The leakage current was found to be marginally de-

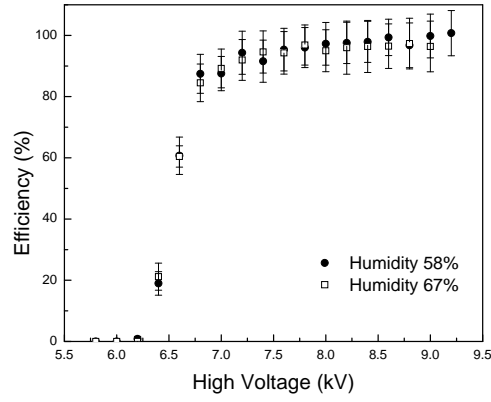


Figure 9: Efficiency versus high voltage for different humidities for silicone coated P-120 grade bakelite RPC.

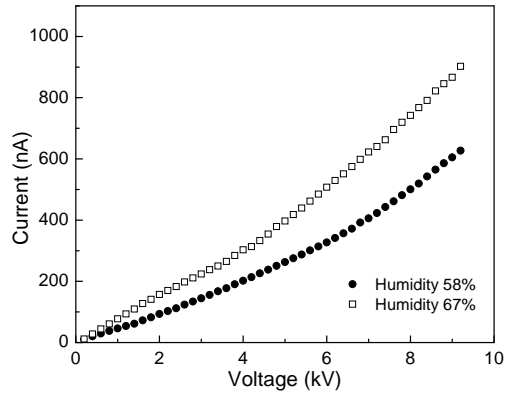


Figure 10: Current versus high voltage for different humidities for silicone coated P-120 grade bakelite RPC.

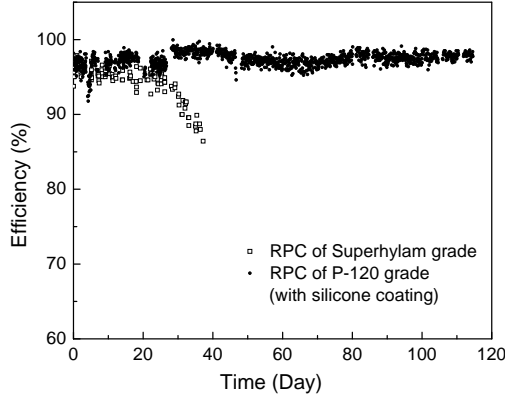


Figure 11: Efficiency as a function of time for two RPC prototypes.

pendent on temperature and humidity, though it has remained steady at ~ 400 nA during the operation.

The superhylam grade RPC has also been tested again after a gap of a few months. It has shown the same higher leakage current ($> 10 \mu\text{A}$) and lower efficiency ($\sim 86\%$) indicating that some intrinsic breakdown of the bulk material may have taken place.

6. Conclusion

We have made a comparative study of bakelite RPCs made from two different grades of bakelite. The RPC, made of superhylam grade bakelite with melamine coated glossy finished surface is found to have a shorter life. On the other hand, the RPC made from P-120 grade bakelite with matt finished surfaces, which are coated with a thin layer of viscous silicone fluid, are found to work steadily for more than 110 days showing a constant efficiency of $> 96\%$ without any degradation. The detector is found to be less immune to variation in humidity which makes it a viable alternative to semiconductive glass based RPC.

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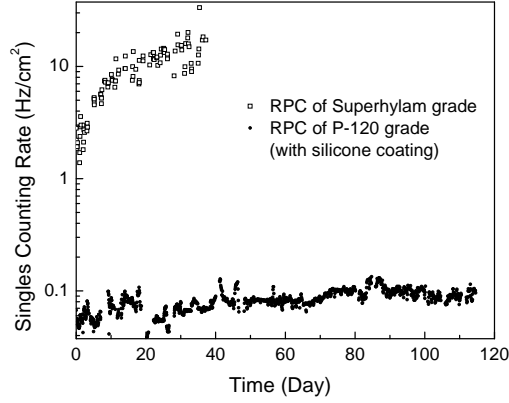


Figure 12: The single counting rate as a function of time for the two RPC prototypes.

of the detectors, and Mr. Ganesh Das of VECC for meticulously fabricating the detectors. Finally we acknowledge the help received from the scientific staff of Electronics Workshop Facility of SINP for building the gas flow control and delivery system of the gas mixing unit used in this study.

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